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UNITED STATES
NAVAL POSTGRADUATE SCHOOL

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THESIS

ANALYSIS OF RADIOACTIVE GAS GENERATED BY THE AGN-201 REACTOR AT THE UNITED STATES NAVAL POSTGRADUATE SCHOOL

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by

Charles C. Grissom

Major, United States Army

Submitted in partial fulfillment of
the requirements for the degree of

MASTER OF SCIENCE
IN
PHYSICS

United States Naval Postgraduate School
Monterey, California

1 9 6 5

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from the

United States Naval Postgraduate School

John D. Riggin

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D. E. Rivell

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ABSTRACT

The principal radioactive constituents of the core gas generated by the AGN-201 reactor at the United States Naval Postgraduate School were identified by an analysis of the spectrum of the emitted gamma-rays and by half-life analysis. Also the absolute activities at various times following generation were determined. A scintillation spectrometer with a gating capability was used. Only those radioisotopes still existing in significant quantities twelve hours after generation were identified since sampling the gas at an earlier time was, for the most part, impractical. The results indicate that the radioisotopes Xe^{133} and Xe^{135} are the only ones present in significant quantities. Their combined specific activities varied from 1.7×10^{-1} to 1.3×10^{-2} microcuries per cc of gas depending upon the age of the gas. It was determined that an upper limit of approximately 0.2% of the activity might be due to the presence of radioactive iodine.

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1. Introduction

When in operation, the AGN-201 reactor at the United States Naval Postgraduate School generates gas in the core and rods. This gas increases the internal pressure and as a result must be bled off periodically and vented into the atmosphere. Most of the gas at the time of venting is presumed to be hydrogen. However, a small percentage of it (about one part in 10^{12}) consists of radioactive elements. Because it is necessary to vent the gas into the local atmosphere while it is still radioactive (generally two to seven days after production), it was required that a qualitative and quantitative analysis of the radioactive constituents of the gas be made. It is the purpose of this paper to discuss the experimental procedures and equipment used in making this analysis and the results obtained.

There are two principal means by which activities of radioactive gases can be studied [4]. The first is to remove the gas from the source in a uniformly flowing system and collect the radioactive decay products of the gases. The second is to collect the active gas in a container and handle it directly. In making this study, the latter method was employed since it was better suited to the physical arrangement of the reactor gas venting system and did not require making any modifications to the gas manifold valving arrangement. The gas usually is not vented any sooner than 48 hours after production since it requires from one to two days for the pressure to build up. Therefore, it was considered that the radioactivity remaining after this long a period of time would be sufficiently long-lived to permit the use of this procedure.

Each time the gas was sampled a gamma-ray spectrum analysis was made on it to locate the energy photo-peaks. These peaks were singled out and decay data were obtained for each of them. From these data the decay constant and half-life of each peak was determined. Comparison of the gamma-ray energies and half lives was made with those of known fission products and conclusions were drawn as to the radioisotopes present in the gas.

The efficiency of the detector was determined for gamma-ray energies of various known sources. Then, by interpolation and extrapolation the efficiency of the detector was estimated for each of the gamma energies predominant in the gas. Relating the efficiency to the number of counts per unit time occurring in a photo-peak provided a means of determining the absolute counting rate of an identified gamma energy. With this information the activity of radioactive gas per unit volume of reactor gas was determined.

2. Equipment and Calibration

a. Scintillation Detector and Preamplifier

A 3" x 3" NaI(Tl) crystal was used. The crystal was a part of a Harshaw type 12S12 scintillation detector which is an integral assembly incorporating a photomultiplier tube all encased in a thin aluminum shell. The scintillation detector assembly was connected to a preamplifier which relayed the signal to the input of a linear amplifier or multi-channel pulse height analyzer as required.

b. Power Supplies

The high voltage for the photomultiplier tube was furnished by a regulated power supply with a manufacturer's specified stability rating against line voltage changes of 3 PPM per volt in the 105-125 volt range with an output voltage of 1000 volts. The preamplifier power was supplied by a stabilized power supply unit in the linear amplifier.

c. Multi-Channel Analyzer

The multi-channel analyzer, an RCL Model 20607, had 128 channels which could be used in various groupings. For purposes of this experiment all 128 channels were used simultaneously. The device was equipped with a cathode ray tube which made possible the visual presentation of the differential spectrum and also the number of pulses recorded in each channel. A Polaroid camera mounted on the face of the tube provided a rapid means of making a permanent record of both the spectrum and counts per channel. Because of gain and voltage fluctuations, this equipment was used primarily as a guide in indicating what to look for rather than providing data for analysis.

d. Single-Channel Analyzer

A Hamner single-channel analyzer was used. It combines the features of a non-overload linear amplifier and an integral-differential discriminator. It was equipped with a voltage control, the "E" dial, which permitted the adjustment of the threshold voltage below which the discriminator rejected all incoming pulses. Also, there was a "ΔE" dial which required that incoming pulses lie between E and E + ΔE in order to be passed on and recorded by a decade scaler which was connected to the output of the discriminator.

e. Sample Bottles

Four sample bottles were used. One was made of brass and two were made of copper. Each of these had a volume of approximately 180 cc (Figure 3). The fourth one was made of aluminum and had a volume of approximately 500 cc. The three smaller bottles were sealed at one end and had a valve and cap at the other end. The valve was such that it could be mated to the reactor gas manifold system. The large bottle had valves at both ends, and one end could be removed thereby permitting the filling of the bottle with activated charcoal.

f. Calibration

Both analyzers were calibrated for energy determination using the gamma-ray photo-peaks from the nuclides Co^{60}(1.17 and 1.33 Mev), Cs^{137}(0.662 Mev), Au^{198}(0.411 Mev), and Sm^{153}(0.103 Mev). To keep from unduly compressing the spectrum and still at the same time be able to determine the positions of higher-energy gamma-rays, two different calibration settings were employed. This was accomplished by varying the position of the Cs^{137} peak using the coarse

gain control on the amplifier. To ensure that the geometrical relationship between gas sample and crystal remained constant, both the sample bottle and case of the scintillation detector were inscribed and prior to each measurement the scribe marks were aligned.

The amplifier linearity was checked in accordance with the manufacturer's recommendations and found to be satisfactory with the exception of a slight nonlinearity that occurred for voltage settings below 4 volts. It was not deemed necessary to correct this slight deviation. The primary photo-peak employed for day-to-day checks on the calibration was the 0.662 Mev peak of Cs^{137}. One calibration setting was made so that the Cs^{137} photo-peak occurred at an "E" setting of 87 volts on the single channel analyzer. For the second calibration setting the photo-peak was positioned so that it occurred at an "E" setting of 24 volts. The same type of calibration was applied to the multi-channel analyzer except, for this device, the photo-peak was positioned in specified channels for the two different calibrations. Prior to each day's measurements and periodically during the day, the calibration was checked. Adjustments, as needed, were made to ensure that the Cs^{137} photo-peak remained in the calibration position. The differential discriminator (ΔE) was also calibrated. It provided a "window" of 9.8 volts in increments of 0.098 volts.

The resolving time of the complete system was determined to be 6.5 $\pm$ 0.5 microseconds.

3. Procedures

a. Gas Sampling

The amount of gas generated is significant only when the reactor is operated at powers in excess of 200 watts for several minutes or more. Particularly significant is the amount of gas produced as a result of the peak power runs of 1000 watts which may last up to about 10 minutes. Therefore, it was decided that the gas would be sampled at various intervals of time following peak power runs. These intervals of time ranged from seven days down to a few hours.

The major problem was to determine what radioactive components existed in the gas in significant quantities at the time of venting and what their relative concentrations were. Therefore, attention was primarily focused on gas that was in excess of 24 hours old.

When sampling, the sampling bottle was connected directly to the reactor manifold system (Figure 1). The line from the core to the manifold was closed and then the manifold, the Geiger chamber connected to the manifold, and the sample bottle were evacuated to approximately 70 cm of Hg below core pressure. The valves leading to the sample bottle and Geiger chamber from the manifold were then closed and the manifold-core valve was opened allowing the core gas to repressurize the manifold. When the manifold system and core pressures were equalized, which occurred very rapidly due to the small volume of the manifold, the manifold-core valve was closed. The manifold-Geiger chamber valve was then opened evacuating the gas from the manifold into the Geiger chamber, leaving the manifold at a pressure several pounds per square inch

below the core pressure. This procedure was performed several times prior to opening the valve leading to the sample bottle. This ensured that the gas sample was, for the most part, composed of gas that had most recently seeped out of the core. After sampling, the gas bottle was left at approximately 3 to 5 cm of Hg below atmospheric pressure. This was done to reduce the possibility of leakage of the gas.

One sample was taken in the large bottle packed with activated charcoal. The general procedure was the same with one exception. The charcoal sample was allowed to remain in contact with the gas in the manifold for approximately two hours during which time five short peak power runs were made. Prior to removing the sample from the manifold the excess gas in the container, i.e., that not adsorbed by the charcoal, was removed.

b. Gamma-Ray Spectrum Analysis

Immediately following sampling, a gamma-ray spectrum was observed visually. The 128-channel pulse-height analyzer was used for this purpose. It is equipped with a cathode-ray tube which makes possible the displaying of the differential spectrum while it is being formed. In addition a picture of the curve was made for future reference. Both previously determined calibration settings for the Cs^{137} photo-peak were used providing a spectral range of from 0 to 2.0 Mev. This provided a rapid means of tentatively locating the energy peaks of the radioactive components of the gas. Following this operation, a second spectral curve was obtained, using the calibrated single-channel amplifier and pulse-height discriminator. The discriminator unit allowed continuous selection of pulse heights from 0 to 100 volts with the window set

at 1 volt. This provided a range of from 0 to 0.75 Mev at the lower calibration setting and 0 to 2.7 Mev for the upper calibration setting. Due to the lack of activity at the higher energies, it was not necessary to use the upper setting. The data obtained were used to plot a curve depicting the energy spectrum of the gas (Figure 2) and from this curve the window width of the various peaks was determined. A sketch of the positioning of the gas sample to detector is shown in Figure 3. The "E" dial of the analyzer was then set at the lower end of the window and the "ΔE" dial was adjusted so that the window included only one specified peak. This was done in turn for all peaks and the data obtained were used for plotting decay curves (Figures 4-6) and determining the half-lives.

c. Determination of Half-Lives

The half-life and decay constant were determined graphically for each component and with the aid of a DCD 1604 computer. The computer program used was FRANTIC II, by P. C. Rogers of M.I.T. [6]. Appendix I contains a brief discussion of the FRANTIC program and a sample output.

d. Determination of Detection Efficiency

To determine the intensity of gamma radiation emitted from a source, it is necessary to know: 1) Ω, the effective fractional solid angle for the particular geometry employed, 2) P_a, the probability that the gamma-ray will not be absorbed between the source and detector, and 3) E_f, the probability that an incident gamma-ray will cause an event in the full energy-peak. Given these parameters and the area under the energy-peak as a count rate and one can determine $\dot{N}_a$, the absolute count rate of the gamma-rays of a given

energy emitted by a source, from

$$\dot{N}_a = \frac{\dot{N}_p}{(\Omega)(E_f)(P_a)}$$

where $\dot{N}_p$ represents the area under the peak in counts per unit time.

For this work the $\dot{N}_a$ values were obtained for three sources, Cs^{137}(0.662 Mev), I^{131}(0.36 Mev), and Sm^{153}(0.103 Mev). The procedure employed was to first determine the absolute disintegration rate of these calibration sources. For this purpose a proportional counter for which an efficiency curve had been previously determined was used to count the beta emissions of the calibration sources. Once the absolute disintegration rate of the calibration sources was known, it was possible to determine that portion of the disintegrations that would evidence themselves by the emission of a gamma-ray whose energy should be detected by the scintillation detector. To determine this fraction the branching ratios of accepted decay schemes were used.

Each source was then placed inside a sample bottle which had its top removed, and, for several positions within the bottle a record was made of the total counts per unit time occurring within the full energy peak. From these data an average counting rate for the bottle for each source was determined. The values obtained included not only the effect of the detection efficiency, but also, the effects of geometry and absorption. The final efficiency for a given source can then be expressed as

$$E_t = E_f \Omega P_a = \frac{\dot{N}_p}{\dot{N}_a} .$$

The E_t's of the identified and suspected gamma-rays in the reactor

gas were then estimated by interpolation between, or extrapolation from these known values. These efficiencies were then used to determine the absolute counting rates of the predominant gamma-rays.

The spectrum of the gas involved gamma-rays of several energies. Therefore, it was a summation of the responses of the detector to the individual gamma-rays. In order to obtain as much accuracy as possible in determining the intensities of the various energies it was necessary to break the gross spectrum down into its components from which the area of the full-energy peaks was then determined.

In addition to determining the activities of the identified gamma-rays, an estimate was made of the amount of radioactive I^{133} and I^{135} present.

4. Results and Conclusions

a. Results

In all cases the gamma-ray energy spectra showed the presence of only three predominant energy peaks. These peaks occurred at 0.08, 0.25, and 0.6 Mev. These energies correspond to the gamma-rays emitted by Xe^{133}(0.081 Mev), Xe^{133m}(0.233 Mev), and Xe^{135}(0.61 & 0.25 Mev). Figure 2 shows the gamma-ray spectra for three samples. Curve A is of gas sampled 12 hours after four short peak power runs. Curve B is of gas sampled 24 hours after the same number of short peak power runs, and Curve C is of gas sampled under the same conditions 48 hours after four short peak power runs. These curves correspond, as far as the energy of the peaks are concerned, extremely well with the gamma-ray spectrum for pure Xe^{133} and Xe^{135}, a plot of which is shown in Figure 7.

An analysis of the decay data provided the following results. The half-lives, determined graphically, have an accuracy of approximately $\pm$ 10%. Those determined by the computer have the standard deviation indicated.

Half-Lives in Hours by Graphical Analysis

	Peak 1	Peak 2	Peak 3
	126	8.7 & 58	9.4
	123	9.2 & 53	8.2
	121	6.9* & 58	8.8
	122	9.1 & 48	
	126	9.7 & 57	
	124		
Mean:	124	9.2 & 54	8.8

* Not included in mean

Half-Lives in Hours by Computer Program

Peak 1	Peak 2	Peak 3
127.8 ± 0.8	8.7 ± 0.0 49.6 ± 2.2	9.3 ± 0.1
121.9 ± 1.0	6.7* ± 0.8 58.5 ± 2.1	8.2 ± 0.0
119.6 ± 1.1	8.8 ± 0.1 51.5 ± 2.9	9.0 ± 0.3
125.7 ± 6.3	9.7 ± 0.1 49.6 ± 8.9	
124.5 ± 1.1	8.7 ± 0.1 50.8 ± 2.2	
125.3 ± 1.4		
Mean:		
124.1 ± 1.9	9.0 ± 0.1 52.0 ± 3.7	8.8 ± 0.1

* Not included in mean

The reason for the difference in the number of half-life determinations made on each peak is due to the difference in the activity of the gas sample. Several samples did not have sufficient activity to determine decay curve data for Peak 3.

The detection efficiencies for the energies of the three peaks and for an assumed peak at an energy of 0.53 Mev are shown below. This latter peak corresponds to a maximum possible activity due to either I^{133} or the decay of I^{135} into Xe^{135m} and the almost immediate subsequent emission of a gamma-ray with an energy of 0.53 Mev, or a combination of these two isotopes.

Energy	Detection Efficiency
0.08 Mev	0.4%
0.23 to 0.25 Mev	4.2%
0.53 Mev	4.5%
0.6 Mev	4.3%

It is estimated that the error involved in determining the detection efficiencies is not in excess of 30%.

Using these efficiencies, the absolute counting rate for each peak and for the assumed peak for each of three different sampling intervals were determined to be as shown below.

Time Interval Following Peak Power Runs (hours)	Energy (Mev)	Absolute Counting Rate (cpm)	Activity ($\mu\mu$c/ml)
12	0.08	40.0×10^6	0.10
	0.25	28.0×10^6	0.070
	0.53	0.084×10^6	0.00021
	0.6	0.48×10^6	0.0013
24	0.08	6.9×10^6	0.017
	0.25	5.1×10^6	0.013
	0.53	0.082×10^6	0.00021
	0.6	0.17×10^6	0.00043
48	0.08	3.4×10^6	0.0085
	0.25	1.7×10^6	0.0043
	0.53	0.01×10^6	0.000025
	0.6	0.028×10^6	0.000070

b. Conclusions

The results of the gamma-ray spectrum analysis indicate the predominant presence of energy peaks at 0.08, 0.25, and 0.6 Mev with half lives of approximately 124, 9.1 & 53, and 8.8 hours, respectively. The only gamma-ray emitted by Xe^{133} has an energy of 0.081 Mev with a half life of 124.8 hours corresponding to Peak 1. Xe^{133m} emits a gamma-ray with an energy of 0.233 and has a half life of 55.2 hours. This corresponds to Peak 2. However, the parent I^{133} decays into approximately 97.6% Xe^{133} and only 2.4% Xe^{133m}. Therefore, the contribution of the Xe^{133m} to Peak 2 is relatively small. Xe^{135} in excess of 97% of the time decays to Cs^{135} with the emission of an electron and an associated gamma-ray with an energy of 0.25 Mev with a half-life of 9.2 hours. This corresponds to the more active part of Peak 2. Something less than

3% of the time the associated gamma-ray has an energy of 0.61 Mev with the same half life and this corresponds to Peak 3.

From the absolute counting rate it is determined that the activity of the gas ranges from 1.7×10^{-1} to 1.3×10^{-2} microcuries per cc of gas at approximately 70^{o}F and 70 cm of Hg for gas which ranges in age from 12 to 48 hours. Of this amount, the maximum activity due to the presence of any radioactive iodine is less than 0.2% of the total activity. At 48 hours approximately 70% of the activity is due to the presence of Xe^{133} and its isomer and approximately 30% of the activity occurs as a result of Xe^{135} being present.

It may therefore be concluded that the primary radioactive constituents of the gas generated by the reactor 12 hours or more after generation are Xe^{133} and its isomer and Xe^{135}. The presence of all other radioactive isotopes is insignificantly small.

5. Acknowledgements

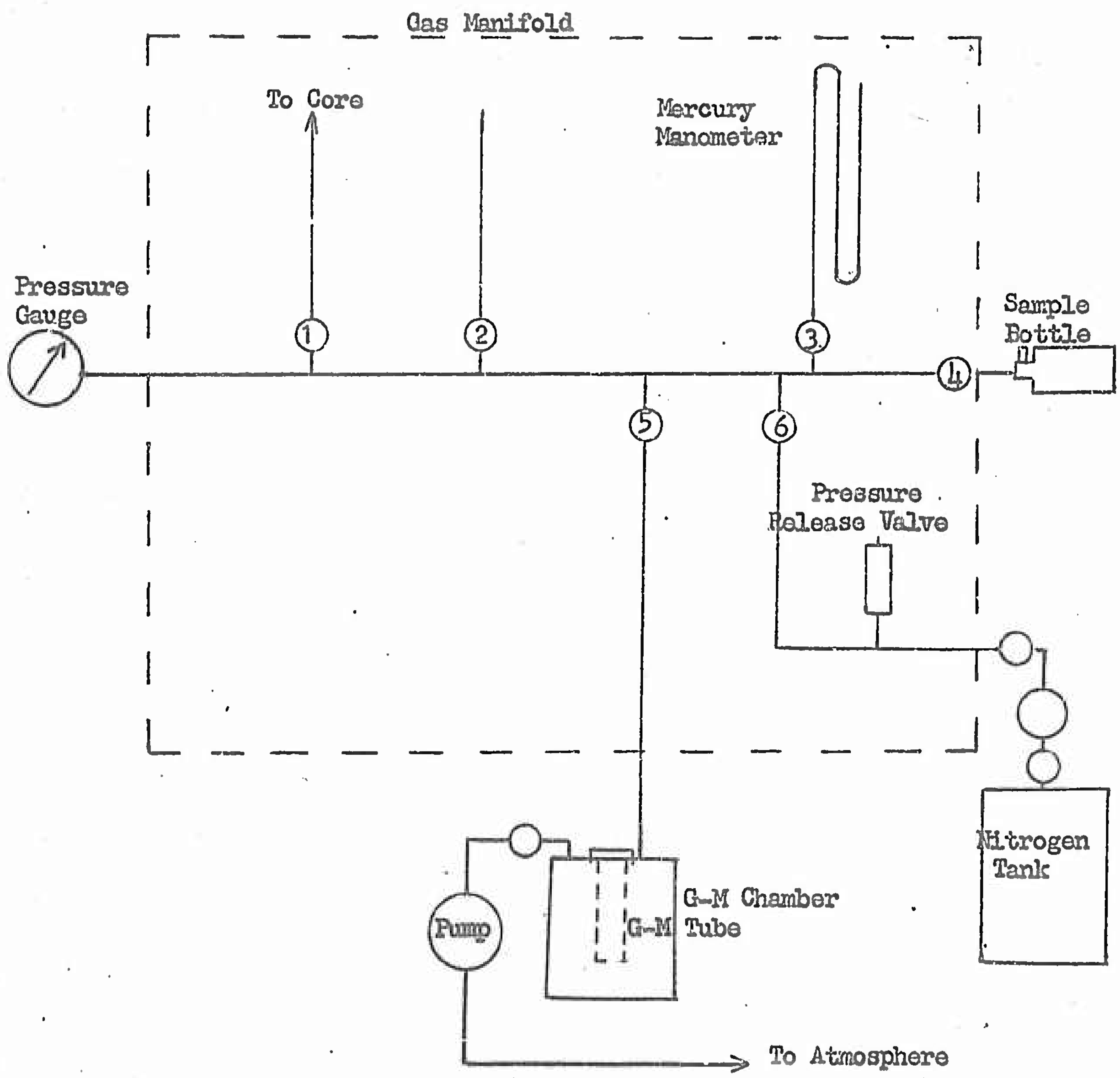
The author wishes to express his appreciation to Professor E. A. Milne who supervised the sampling of the gas and was most generous in his counseling and to Professor J. D. Riggin, thesis advisor, who gave freely of his time in providing encouragement and guidance.

FIGURE 1

Diagram of Reactor Gas Manifold

Numbered Valves:

1. Core valve
2. Auxiliary gas sampling tube valve
3. Manometer valve
4. Gas sampling valve
5. G-M chamber valve
6. Nitrogen pressurizing valve



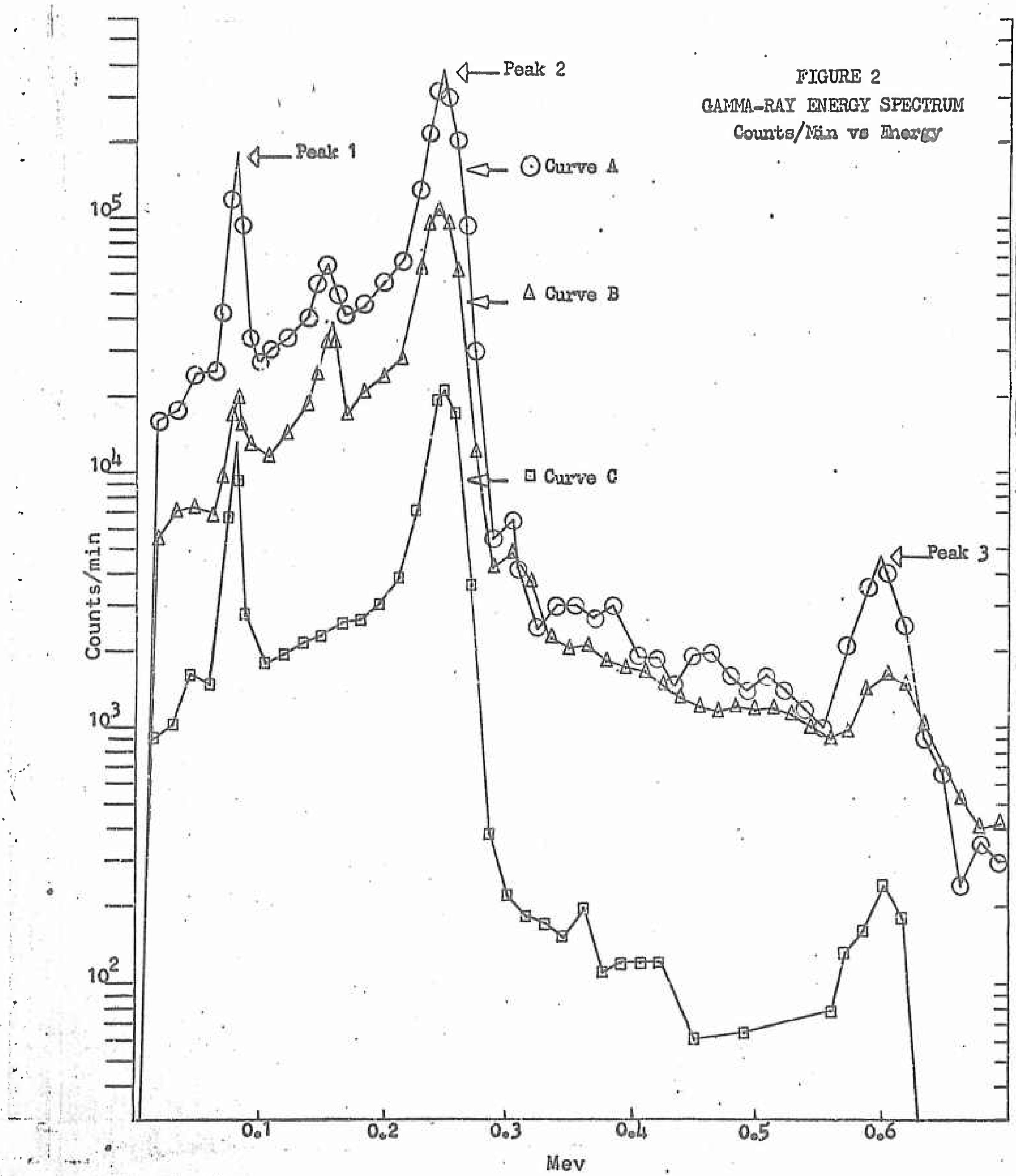
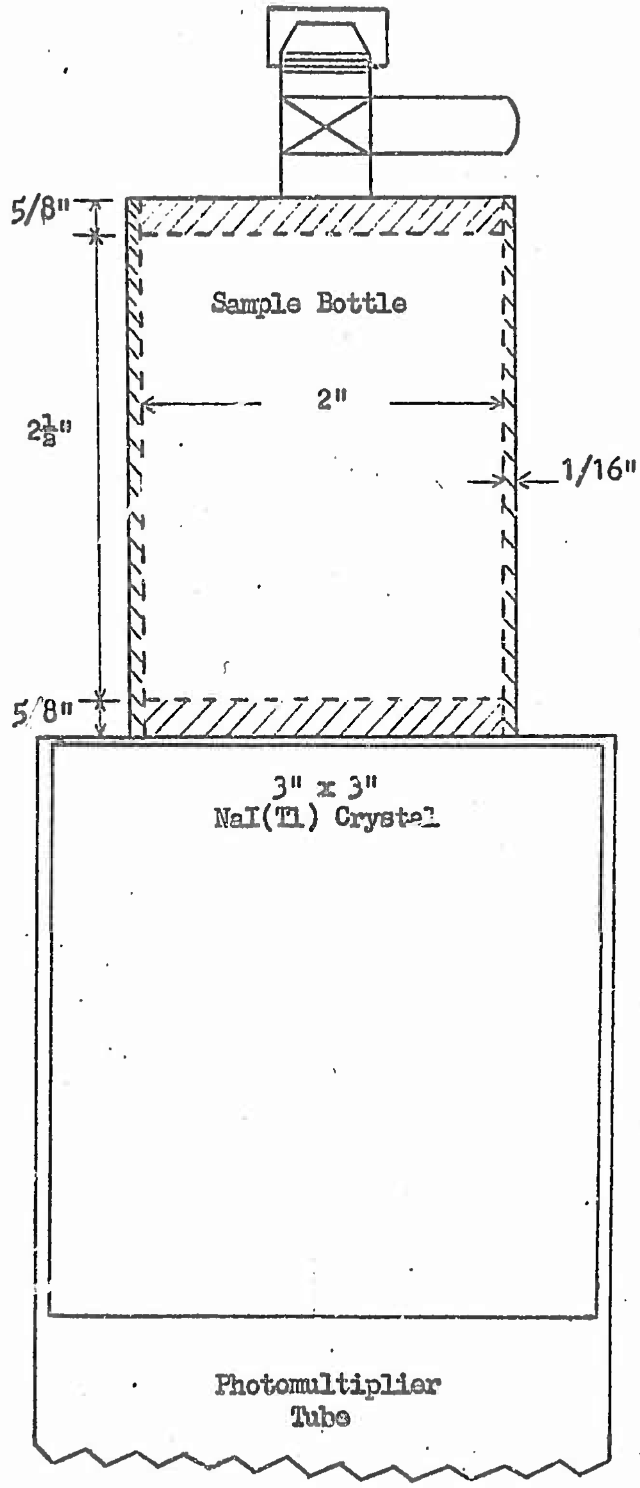


FIGURE 2
GAMMA-RAY ENERGY SPECTRUM
Counts/Min vs Energy
Peak 1
Peak 2
Peak 3
Curve A
Curve B
Curve C
Counts/min
Mev
10^5
10^4
10^3
10^2
0.1
0.2
0.3
0.4
0.5
0.6

FIGURE 3

Diagram of Scintillation Detector and Sample Bottle



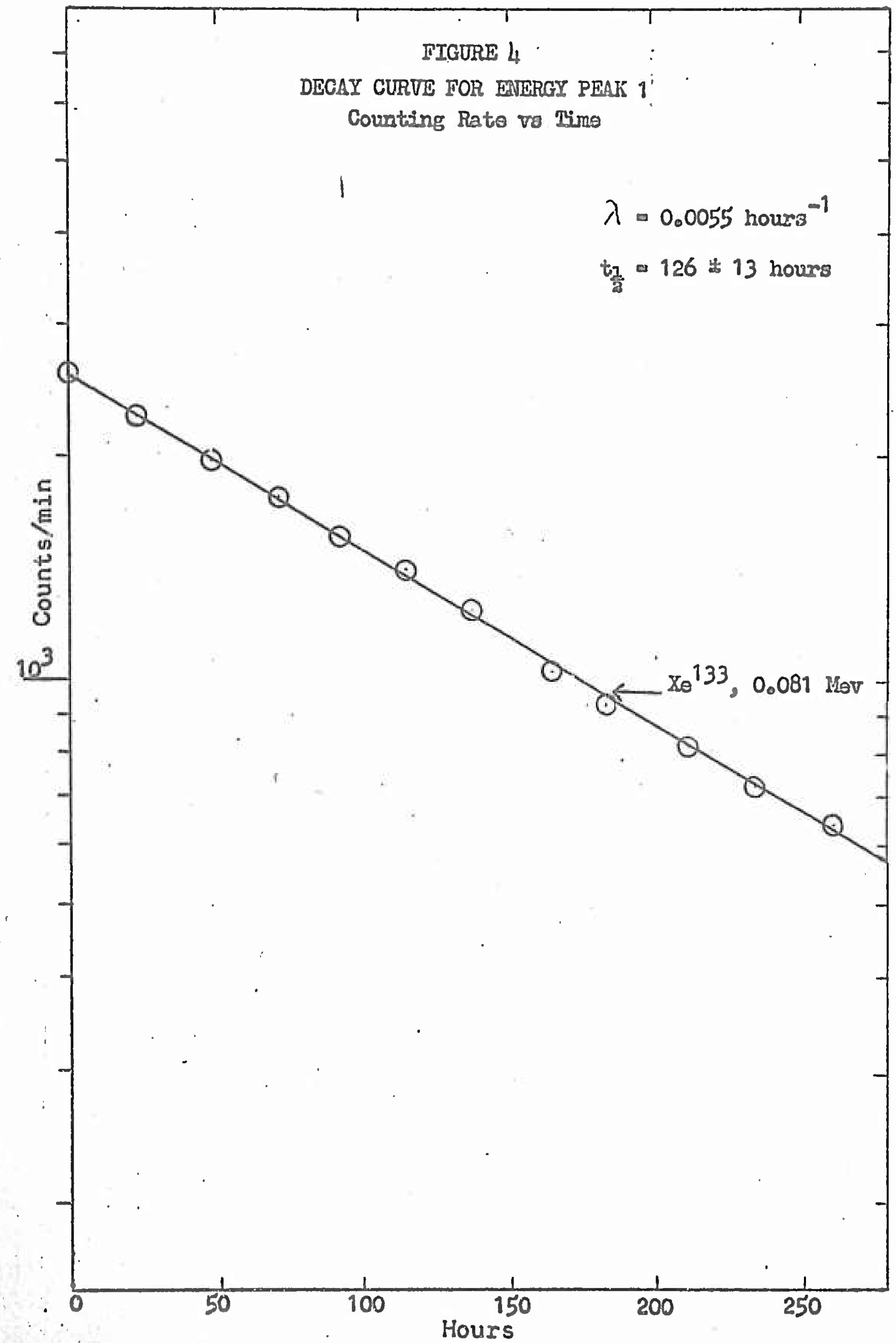


FIGURE 4
DECAY CURVE FOR ENERGY PEAK 1
Counting Rate vs Time
λ = 0.0055 hours⁻¹
t½ = 126 ± 13 hours
Xe133, 0.081 Mev
Counts/min
10³
0
50
100
150
200
250
Hours

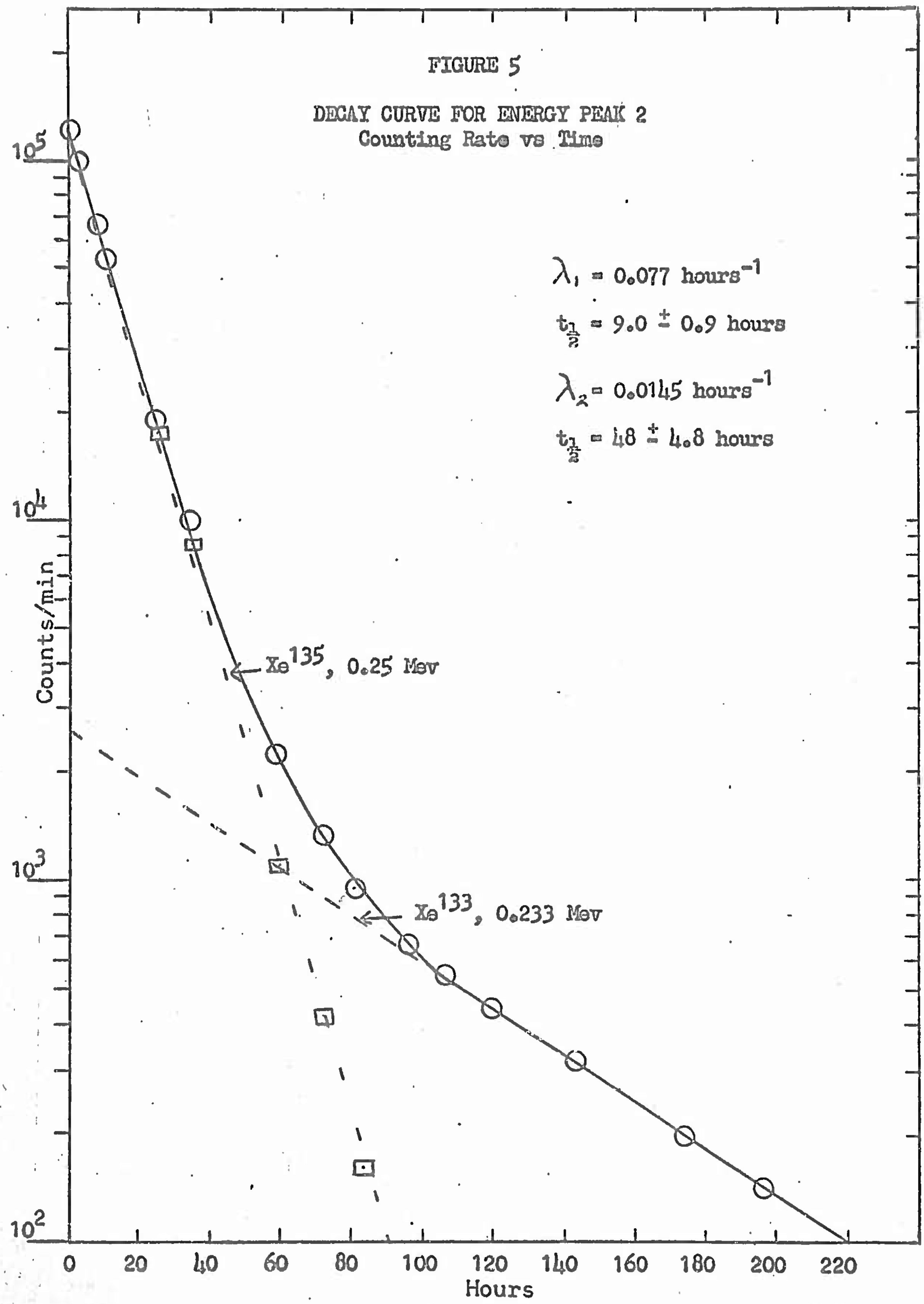


FIGURE 5
DECAY CURVE FOR ENERGY PEAK 2
Counting Rate vs Time
λ_1 = 0.077 hours^{-1}
$t_{\frac{1}{2}}$ = 9.0 ± 0.9 hours
λ_2 = 0.0145 hours^{-1}
$t_{\frac{1}{2}}$ = 48 ± 4.8 hours
Xe^{135}, 0.25 Mev
Xe^{133}, 0.233 Mev
Counts/min
10^5
10^4
10^3
10^2
0
20
40
60
80
100
120
140
160
180
200
220
Hours

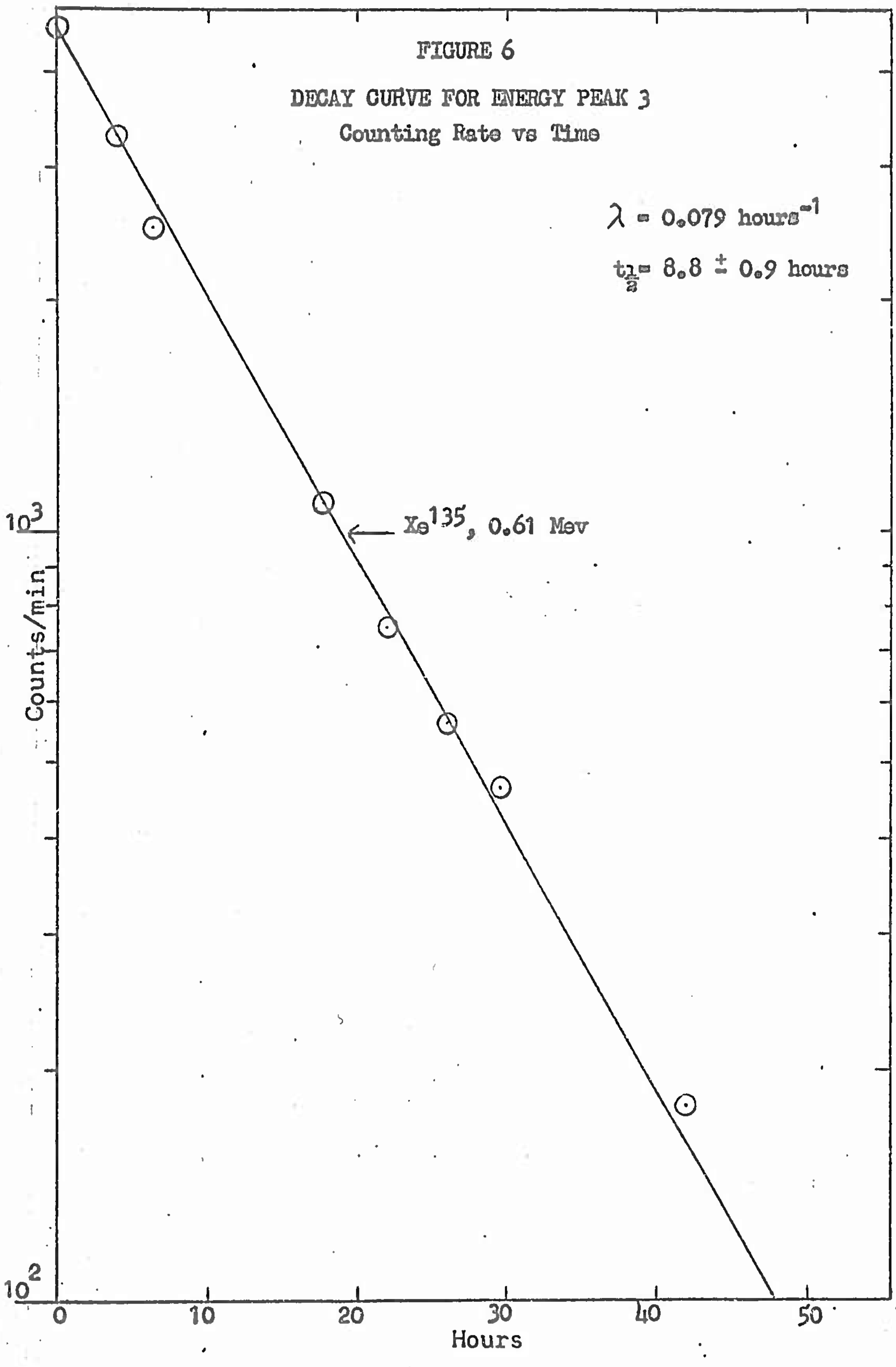


FIGURE 6
DECAY CURVE FOR ENERGY PEAK 3
Counting Rate vs Time
λ = 0.079 hours⁻¹
t½ = 8.8 ± 0.9 hours
Xe135, 0.61 Mev
10³
10²
Counts/min
0
10
20
30
40
50
Hours

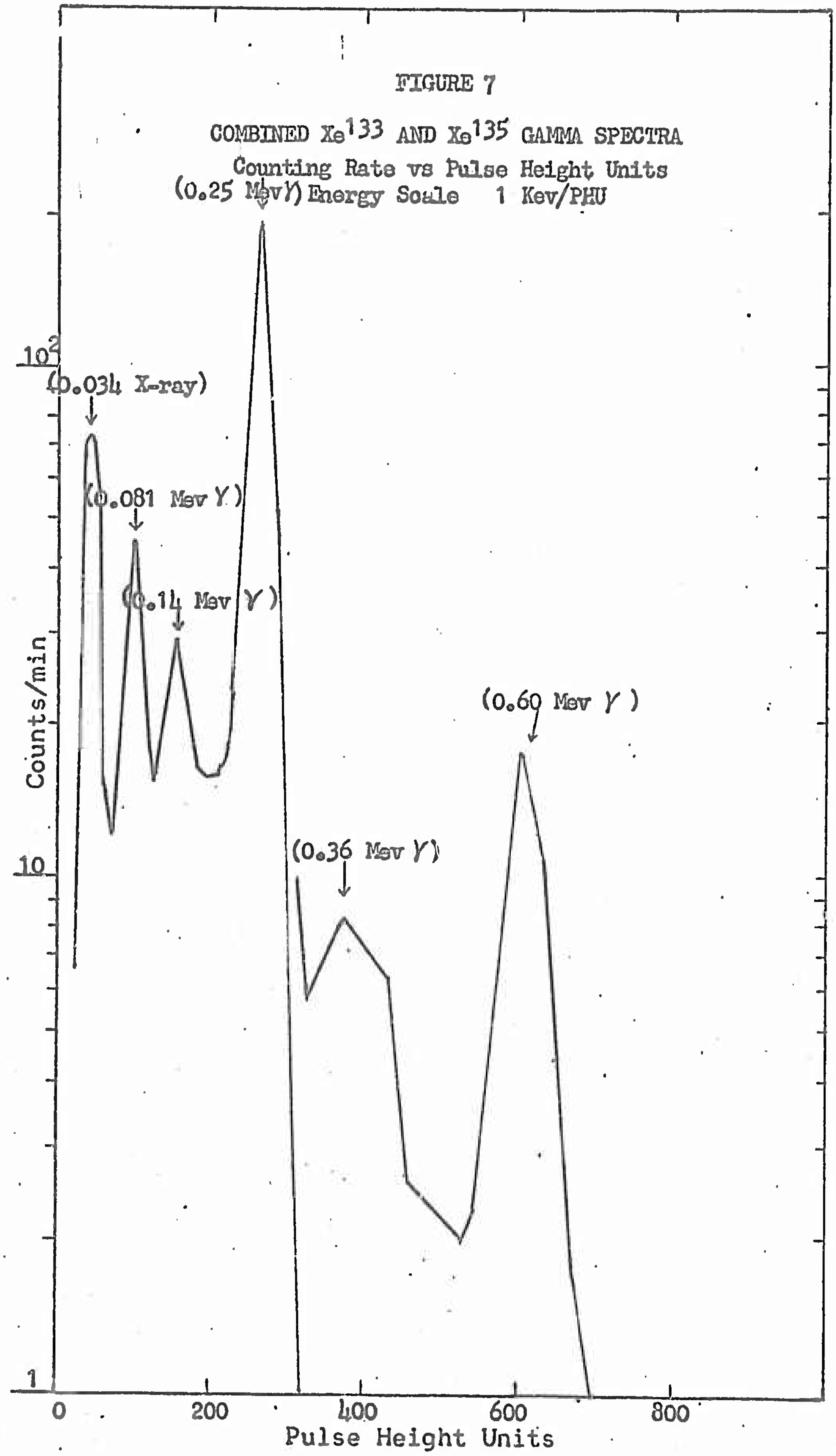


FIGURE 7
COMBINED Xe^{133} AND Xe^{135} GAMMA SPECTRA
Counting Rate vs Pulse Height Units
Energy Scale 1 Kev/PHU
(0.25 Mev γ)
(0.034 X-ray)
(0.081 Mev γ)
(0.14 Mev γ)
(0.36 Mev γ)
(0.60 Mev γ)
10^2
10
1
Counts/min
0
200
400
600
800
Pulse Height Units

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APPENDIX I

FRANTIC COMPUTER PROGRAM

The data for the decay of the energy peaks were analyzed using the computer program called FRANTIC[1]. This program is written in Fortran for use with the CDC 1604 computer. The program processes raw counting data and fits these data by an iterative least-squares analysis to equations for multiple exponential growth and decay curves. In the process the input data points are corrected for dead time and background. Each point is then normalized as desired to yield the correct data for the n^{th} data point.

[1]For further details refer to item (6), Bibliography.

RGS 4 PEAK 2

```
RGS 4   P 2 = FRANTIC IDENTIFICATION                       .00000 = TAU (TIME TO COUNTING)
          2 = NUMBER OF COMPONENTS                         .01000 = TAUD (DEAD TIME FACTOR IN MICRO TIME UNITS)
          0 = NUMBER OF PARAMETERS HELD FIXED              .00100 = ERROR IN DEAD TIME FACTOR (MICRO TIME UNITS)
         12 = NUMBER OF DATA POINTS                        .00000 = BACKGROUND
          8 = DEGREES OF FREEDOM                           .00000 = UNCERTAINTY IN TIMING INTERVAL IN TIME UNITS
          9 = ITERATIONS                                  1.00000 = DATA SCALE FACTOR
          2 = STATISTICAL WEIGHTS
                                                          1.00000 = NORMALIZATION FACTOR
          2 = NUMBER OF POINTS (*) DEVIATING MORE THAN 2 SIGMA
     .01059 = LEAST SQUARES DETERMINANT
     .00025 = DELTA VARIANCE OF FIT X 10(6)
    2.75307 = WEIGHTED VARIANCE OF FIT
   22.15448 = CHI SQUARE
```

A(ZERO)	SIGMA	LAMBDA	SIGMA	N(ORIGINAL)	SIGMA	HALF LIFE	SIGMA
3168273.893	31420.740	.086608947	.001477892	36582370.	781996.	8.0032	.1366
179070.067	13551.005	.013453665	.000750834	13310133.	1256237.	51.5211	2.9060

ORIGINAL ESTIMATES

3143710.970	.079500000
138543.413	.011900000

25.

ANALYSIS OF THE DEVIATIONS

```
        0    0    0    0    0    0    1    1    1    2    1    1    1    1    2    1    0    0    0    0    0    0
SIGMA -XS -4.5 -4.0 3.5 -3.0 -2.5 -2.0  -1   .5 -1.0 -0.5 -0.0  0.0  0.5  1.0  1.5  2.0  2.5  3.0  3.5  4.0  4.5  XS
```

	BEGINNING TIME	INTERVAL TIME	ORIGINAL COUNTS	CORRECTED DATA	CALCULATED RATE	INSTANTANEOUS RATE	WEIGHTS X 10(3)	DELTA RATE
1	.000	.017	53984.0	3347465.893	3345038.312	3345038.037	.00000	2427.582
2	25.900	.017	7589.0	457422.840	462349.180	462349.159	.00004	-4926.341
3	46.600	.017	2609.0	156785.438	151590.717	151590.712	.00011	5194.721
4	48.700	.017	2425.0	145712.017	139623.735	139623.731	.00011	6088.282
* 5	51.700	.017	2076.0	124715.350	125274.447	125274.443	.00013	-559.096 *
6	54.700	.017	1814.0	108958.595	113512.331	113512.329	.00015	-4553.736
7	56.700	.017	1742.0	104629.363	106823.877	106823.875	.00016	-2194.515
8	70.200	.017	1326.0	79623.352	76876.260	76876.267	.00021	2747.084
9	94.900	.017	790.0	47422.480	50797.517	50797.517	.00035	-3375.037
* 10	117.500	.017	632.3	37934.386	36970.614	36970.614	.00044	963.773 *
11	141.100	.017	424.0	25446.475	26841.103	26841.133	.00046	-1394.620
12	170.300	.017	332.0	19923.970	18111.999	18111.999	.00084	1811.071

TIME, 1 MINUTES AND 47 SECONDS

Sample Computer Output